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(54) **Cardable hydrophobic polypropylene fiber.**

(57) A method for treating the surface of hydrophobic polyolefin-containing fibers to improve their lubricity and antistatic properties comprises applying a liquid lubricating finish to the surface of an extruded polyolefin-containing fiber or filament, in which the finish is either (1) a polyol, (2) a water-soluble ester or polyester obtained by reacting the polyol with a fatty acid having up to 6 carbon atoms, or (3) a glycol obtained by reacting the polyol with ethylene oxide or a combination of ethylene oxide and propylene oxide, and then mechanically processing the fiber or filament, the finish providing lubricity for the processing operations and lacking sufficient lipophilic substituents to produce significant surfactant properties; and the use of the said finish.

EP 0 516 412 A2

This invention relates to a method for treating the surface of hydrophobic polyolefin-containing fibers to improve their lubricity and antistatic properties and to facilitate processing them into hydrophobic nonwoven articles.

5 In the usual processing operations, such as spinning, stretching, crimping, and carding, for manufacturing nonwoven material from conventionally bonded hydrophobic webs of polyolefin-containing staple fiber, untreated hydrophobic web quickly becomes unworkable due to friction and accumulated static charges.

10 It is known, for instance from U.S. Patent 3,341,451, that antistatic agents containing salts of phosphoric acid esters change fiber surface properties sufficiently to facilitate conventional fiber processing, but such antistatic treatments also make the fibers and the nonwoven material produced substantially more hydrophilic than the untreated spun fiber.

15 It is also known, for instance from U.S. Patent 3,423,314, that dimethylpolysiloxane fluids, which are effective lubricants for synthetic fibers, do not provide static protection even when modified with antistatic agents known as of the date of the patent.

20 It is also known, for instance from U.S. Patent 4,938,858, that surface treatment of polyolefin-containing fibers with a neutralized phosphoric acid ester, followed by treatment with a poly(dialkylsiloxane), preferably after any crimping step, increases their lubricity and anti-static properties and facilitates processing into hydrophobic nonwoven articles, while retaining a level of hydrophobicity that is acceptable for use as the layer of hydrophobic material in products used for personal hygiene, such as catamenial devices, disposable diapers, incontinence pads and the like, that have a fluid-absorbent core and layer of hydrophobic material that isolates fluids already absorbed in the core.

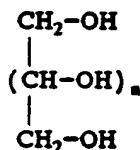
25 There is a need for an improved method for imparting anti-static properties and lubricity to polyolefin-containing hydrophobic fibers or filaments to facilitate processing with less interference with their hydrophobicity or bonding properties.

30 According to the invention, a method for treating the surface of hydrophobic polyolefin-containing fibers to improve their lubricity and antistatic properties comprises applying a liquid lubricating finish to the surface of an extruded polyolefin-containing fiber or filament, characterized in that the finish is selected from

(1) a polyol having the formula:



or



35 in which R is an alkyl group having 1 to 4 carbon atoms; m is 0 to 3 and n is 0 to 4;

(2) a water-soluble ester or polyester obtained by reacting the said polyol with a fatty acid having up to 6 carbon atoms in a linear or branched chain;

40 (3) a glycol obtained by reacting the said polyol with ethylene oxide;

and then mechanically processing the fiber or filament, the finish providing lubricity for the processing operations and lacking sufficient lipophilic substituents to produce a significant surfactant effect on the fiber or filament.

45 After the finish treatment, the processing steps may involve crimping, optional overfinishing, cutting (normally to a 3/4"-1-1/2" length) or carding (to form fiber webs). The webs are then conventionally compiled and bonded to obtain a hydrophilic nonwoven material.

Preferably, the finish-treated fiber or filament is washed with water after at least one of the mechanical processing steps to remove at least some of the finish.

50 The preferred polyols of group (1) are glycerol, ethylene glycol, propylene glycol, neopentyl glycol, trimethylolethane, trimethylolpropane and pentaerythritol.

The preferred water-soluble esters or polyesters of group (2) are obtained by reacting the said preferred polyols of group (1) with a fatty acid having up to 6 carbon atoms in a linear or branched chain to obtain esters such as glycerol triacetate, pentaerythritoltracetate, propylene glycol dipropionate, and trimethylolpropane dibutanoate.

55 The preferred glycols of group (3), which may also be glycols capped by one ester group of up to 6 carbon atoms, are obtained by reacting the said preferred polyols with ethylene oxide. Preferred examples of the glycols are polyoxyethylene glycol (POE glycol preferably with a molecular weight of 400 or 2000), POE glycerol, preferably with 10 mols of POE to one mol of glycerol, (referred to as POE (10) glycerol), POE (20) sorbitol,

POE (10) sorbitol dipropionate, and polyethylene glycol (PEG) diacetate, preferably with a molecular weight of 600. More preferred are glycols obtained by reacting the polyols with a combination of ethylene oxide and up to about 20% of propylene oxide to obtain a block or random-type polyoxyalkylene polyol, such as butyl-capped EO/PO polymer (90/10 ratio with an average molecular weight of 1000).

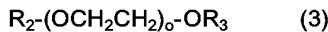
5 Generally, the finishes that can be used in the process according to the invention, whether used as initial spin finishes or as overfinishes, are water soluble finish components having low or limited surfactant properties within the class defined by the formulas:



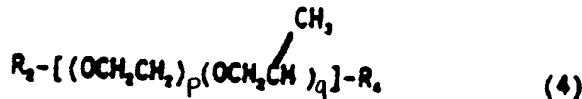
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25 in which each R is hydrogen or a 1-4 carbon alkyl group, inclusive of  $CH_3-$  and  $C_4H_9-$ ;  $R_1$ ,  $R_2$ ,  $R_3$ , and  $R_4$  are individually defined as hydrogen or a straight or branched lower molecular weight acyl, or alkoxy, inclusive of  $CH_3CO-$  to  $CH_3(CH_2)_4CO-$ ,  $CH_3-O-$ ,  $CH_3(CH_2)_5-O-$ ;  $m$  is 0-3;  $n$  is 0-4;  $o$  is 2-50;  $p$  is 2-50;  $q$  is 1-10; and  $p/q$  is at least 4.

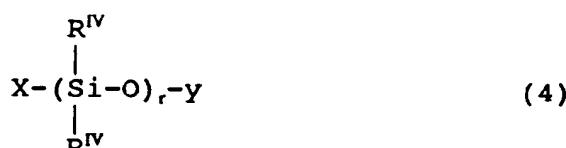
30 The most preferred finishes are polyoxyethylene glycol (POE glycol) 400, POE glycol 2000, POE (10) glycerol (a convenient abbreviation commonly used for ten moles of ethylene oxide to one mol of glycerol), POE (20) sorbitol, POE (10) sorbitol dipropionate, polyethylene glycol (PEG) 600 diacetate, or butyl-capped EO/PO polymer in a 90/10 ratio with an average molecular weight of 1000.

35 The liquid finish (and a conventional amount of antistatic agent if desired) can be applied to the surface of the fiber or filament by any conventional procedure, such as by dipping, spraying or wheel printing, and then dried. It may be the product in neat form, in the form of an aqueous solution (1-99% by weight), or as a water/oil emulsion. Preferably it is carried onto the fiber or filament as an aqueous solution by a roller immediately following a quenching step. The preferred amount applied is about 0.02%-0.8% and more preferably 0.1%-0.5% of the water-soluble finish, based on the total fiber weight.

40 A preferred overfinishing step may comprise applying about .05%-.80%, and preferably 0.1%-0.5% (by weight of fiber), of an overfinish composition at or downstream from a fiber crimping station, the overfinish comprising:

(A) about 0%-65%, by composition weight, of at least one polysiloxane represented by the formula

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50 in which X and y are individually defined as a hydrophobic chemical end group such as a lower alkyl group; Each  $R^{IV}$  is individually defined as a lower alkyl such as a methyl or octyl group; and  $r$  is a positive number within the range of at least about 10 and preferably up to about 50; and

(B) about 35%-100%, by weight of overfinish composition, of at least one neutralized phosphoric acid ester, as an antistatic agent, having the formula

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in which each Alk is individually defined as a lower alkyl group, inclusive of a 1-8 carbon alkyl, preferably a 4 to 8 carbon alkyl such as butyl or octyl;

$\text{R}'$  is an amino group or an alkali metal; and

$s$  and  $t$  are individually positive numbers of not less than about 1, the sum of which is about 3.

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The term "polyolefin-containing fiber or filament" includes continuous, as well as staple (cut) melt spun fiber obtainable from conventionally blended isotactic polypropylene and/or known hydrophobic copolymers thereof with ethylene, 1-butene, 4-methylpentene-1 and the like. The resulting extrudable spun melt preferably has a weight average molecular weight varying from about  $3 \times 10^5$  to about  $5 \times 10^5$ , a molecular weight distribution of about 5.0-8.0, a spun melt flow rate of about 13.0 to about 40 g/10 minutes, and a fiber spin finish temperature within a range of about 220°C-315°C.

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Also included within the definition of polyolefin-containing spun melt are various conventional fiber additives, including pH stabilizers such as calcium stearate, antioxidants, and pigments, such as whiteners and colorants, including  $\text{TiO}_2$ . Generally such additives can vary, in amount, from about 0.05%-3% collectively by weight of spun melt.

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The invention is further illustrated by the following Examples and Tables, in which conventional absorbency testing is used, based on a modified ASTM test Method D-1117-79, in which five (5) grams of the dry treated staple fiber is loosely packed into a 50 CC wire basket, weighed and then placed into a tank of water. After 30 seconds, the basket is removed, drained for 30 seconds and then weighed to measure the amount of water absorbed and percent absorbency calculated on a weight basis.

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#### EXAMPLE 1

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Polypropylene fiber samples S-1, S-2 and S-3 are individually prepared from a conventionally stabilized polypropylene resin batch in flake form having a molecular weight distribution of about 5.0 and a melt flow rate of about 13.0 g/10 minutes.

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Each resin sample is then admixed with .5% by weight of calcium stearate as a conventional pH stabilizer and .1% by weight of titanium dioxide as pigment for sixty (60) minutes in a tumbling blender. The blended flake is then extruded through a 675 circular hole spinnerette at 300°C and the resulting extruded filaments are air quenched at ambient temperature, and an initial spin finish ("A", "B" or "C"), as indicated in Table I, of glycerol and morpholine-neutralized phosphoric acid ester is topically applied to the respective filaments by a roll applicator, to impart about 0.3%-0.5% by dry fiber weight of the initial finish.

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The resulting coated filaments are then drawn to about 2.0-2.4 dpf (grams/9000 meters) and crimped in a conventional steam crimper (100 C), with simultaneous application of the "D" or "E" (Finish D is a morpholine-neutralized phosphoric acid ester alone and Finish E is a 50%/50% ratio of morpholine-neutralised phosphoric acid ester and polydimethylsiloxane, obtained commercially from Union Carbide Corporation as LE-458HS), and applied through steam injection holes in the crimper stuffer box. Control sample S-3 utilised Lurol PP-912, obtained commercially from George A. Goulston Co. of Monroe, NC. as a standard hydrophilic spin finish.

The treated filaments are then dried, cut into 1.5 inch staple, and set aside for conventional absorbency and hydrophobicity testing. The results are reported in Table I.

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#### EXAMPLE 2

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Polypropylene fiber sample S-4, S-5 and S-6 are prepared by tumbling the same batch resin plus identical stabilizer and pigment in the same amount and manner as Example 1, the blended flake then extruded at 295°C. through a 782 circular hole spinnerette, and air quenched at the ambient temperature. The resulting filaments are then topically treated by roll applicator with a 1% aqueous solution of potassium-neutralized phosphoric acid ester as a spin finish to obtain about 0.16% initial filament finish based on dry fiber weight.

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The resulting filaments are then drawn, as before, to about 2.0-2.4 dpf, steam crimped, and an overfinish applied through steam injection holes in the crimper stuffer box to obtain a final finish of about 0.20%-0.50% by weight, the fiber then being dried, cut into 1.5 inch length staple, and set aside for testing. Test results are reported in Table II.

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TABLE I

Sample #	Spin Finish (#3)	Level	Overfinish <sup>(*)</sup>	Total Finish	Total Absorbency
S-1	A.	0.33 <sup>‡</sup>	D.	0.39 <sup>‡</sup>	59%
S-2	B.	0.49 <sup>‡</sup>	E.	1.70 <sup>‡</sup>	575%
S-3 (control)	C.	0.30 <sup>‡</sup>	C.	0.90 <sup>‡</sup>	1382%

(\*) Finish A is a 25<sup>‡</sup>/75<sup>‡</sup> ratio of morpholine-neutralized phosphoric acid ester and glycerol

Finish B is a 17<sup>‡</sup>/83<sup>‡</sup> ratio of morpholine-neutralized phosphoric acid ester and glycerol

Finish C is a commonly used hydrophilic finish for polypropylene identified as Lurol PP 912.

Finish D is a morpholine-neutralized phosphoric acid ester alone.

Finish E is a 50<sup>‡</sup>/50<sup>‡</sup> ratio of morpholine-neutralized phosphoric acid ester and dimethylpolysiloxane.

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TABLE II

Sample #	Spin Finish (%)	Level	Overfinish (*)	Finish	Total Absorbency
S-4	F.		0.16‡	G.	0.21‡
S-5	F.		0.16‡	H.	0.26‡
S-6	F.		0.16‡	I.	0.49‡

(\*) Finish F is Potassium-neutralized phosphoric acid ester.

Finish G is a 50% / 50% ratio of potassium-neutralized phosphoric acid ester and polydimethylsiloxane.

Finish H is a 50% / 50% ratio of potassium-neutralized phosphoric acid ester and polyoxyethylene glycol (400).

Finish I is 17% / 83% ratio of potassium-neutralized phosphoric acid ester and polyoxyethylene glycol (400).

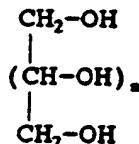
**Claims**

1. A method for treating the surface of hydrophobic polyolefin-containing fibers to improve their lubricity and antistatic properties comprises applying a liquid lubricating finish to the surface of an extruded polyolefin-containing fiber or filament, characterized in that the finish is selected from the group consisting of

5 (1) a polyol having the formula



or



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in which R is an alkyl group having 1 to 4 carbon atoms; m is 0 to 3 and n is 0 to 4;

(2) a water-soluble ester or polyester obtained by reacting a polyol having one of the said formulas with a fatty acid having up to 6 carbon atoms in a linear or branched chain;

(3) a glycol obtained by reacting a polyol having the said formula with ethylene oxide;

20 and then mechanically processing the fiber or filament, the finish providing lubricity for the processing operations and lacking sufficient lipophilic substituents to produce significant surfactant properties.

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2. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in claim 1, further characterized in that the finish is a water-soluble esters or polyester obtained by reacting a polyol of said group (1) with a fatty acid having up to 6 carbon atoms in a linear or branched chain.

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3. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in claim 1, further characterized in that the finish is a glycol obtained by reacting a polyol of the said group 1 with ethylene oxide.

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4. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in claim 3, further characterized in that the finish is a glycol obtained by reacting with ethylene oxide a polyol of the said group 1 that is capped by one ester group of up to 6 carbon atoms.

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5. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in claim 1, further characterized in that the finish is glycerol, ethylene glycol, propylene glycol, neopentyl glycol, trimethylol-ethane, trimethylolpropane, pentaerythritol, or sorbitol.

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6. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in claim 3 or 4, further characterized in that the finish is polyoxyethylene glycol (POE glycol) 400, POE glycol 2000, POE (10) glycerol, POE (20) sorbitol, polyethylene glycol (PEG) 600 diacetate, or POE (10) sorbitol dipropionate.

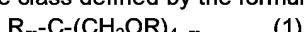
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7. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in claim 1, further characterized in that the finish is a block or random-type polyoxyalkylene polyol obtained by reacting a polyol of the said group 1 with a combination of ethylene oxide and up to about 20% of propylene oxide.

8. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in claim 7, further characterized in that the finish is butyl-capped EO/PO polymer in a 90/10 ratio with an average molecular weight of 1000.

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9. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in any of the preceding claims, further characterized in that the finish comprises a water soluble compound having low or limited surfactant properties within the class defined by the formulas:

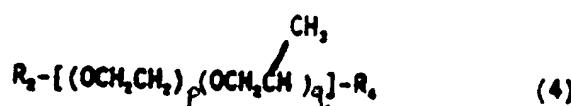


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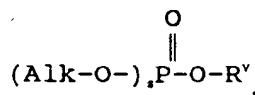
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in which each R is hydrogen or a 1-4 carbon alkyl group, inclusive of  $\text{CH}_3$ - and  $\text{C}_4\text{H}_9$ -;  $\text{R}_1$ ,  $\text{R}_2$ ,  $\text{R}_3$ , and  $\text{R}_4$  are individually defined as hydrogen or a straight or branched lower molecular weight acyl, or alkoxy, inclusive of  $\text{CH}_3\text{CO}-$  to  $\text{CH}_3(\text{CH}_2)_4\text{CO}-$ ,  $\text{CH}_3-\text{O}-$ ,  $\text{CH}_3(\text{CH}_2)_5-\text{O}-$ ;  $m$  is 0-3;  $n$  is 0-4;  $o$  is 2-50;  $p$  is 2-50;  $q$  is 1-10; and  $p/q$  is at least 4.

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10. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in any of the preceding claims, further characterized in that neutralized phosphoric acid ester, as an antistatic agent, having the formula

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in which each Alk is individually defined as a lower alkyl group, inclusive of a 1-8 carbon alkyl, preferably a 4 to 8 carbon alkyl such as butyl or octyl;

$\text{R}^v$  is an amino group or an alkali metal; and

$s$  and  $t$  are individually positive numbers of not less than about 1, the sum of which is about 3, is applied to the fibers.

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11. A method for treating the surface of hydrophobic polyolefin-containing fibers as claimed in claim 10, further characterized in that the antistatic agent is a morpholine-neutralized phosphoric acid ester.

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12. Use of a liquid lubricating finish as defined in any of the preceding claims to treat the surface of a hydrophobic extruded polyolefin-containing fiber or filament prior to mechanically processing the fiber or filament to provide lubricity for the processing operations in the absence of a significant decrease in hydrophobicity.

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**DERWENT-ACC-NO:** 1992-400760

**DERWENT-WEEK:** 200142

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**TITLE:** Treatment of hydrophobic polyolefin-contg. fibres uses finish selected from poly:ol, water-soluble ester of polyester and glycol to improve lubricity and antistatic properties

**INVENTOR:** JOHNSON R W; THEYSON T W

**PATENT-ASSIGNEE:** HERCULES INC [HERC]

**PRIORITY-DATA:** 1991US-706450 (May 28, 1991) ,  
1992US-973583 (November 6, 1992) ,  
1993US-115374 (September 2, 1993)

**PATENT-FAMILY:**

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EP 516412 A2	December 2, 1992	EN
AU 9217209 A	December 3, 1992	EN
BR 9202012 A	January 12, 1993	PT
CA 2069269 A	November 29, 1992	EN
JP 05156571 A	June 22, 1993	JA
TW 205576 A	May 11, 1993	ZH
AU 653403 B	September 29, 1994	EN
EP 516412 A3	September 8, 1993	EN
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EP 516412A2	N/A	1992EP- 304839	May 28, 1992
CA 2069269A	N/A	1992CA- 2069269	May 22, 1992
CA 2069269C	N/A	1992CA- 2069269	May 22, 1992
IL 101987A	N/A	1992IL- 101987	May 25, 1992
AU 9217209A	N/A	1992AU- 017209	May 27, 1992
AU 653403B	N/A	1992AU- 017209	May 27, 1992
BR 9202012A	N/A	1992BR- 002012	May 27, 1992
EP 516412A3	N/A	1992EP- 304839	May 28, 1992
JP 05156571A	N/A	1992JP- 137046	May 28, 1992
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CIPS	D04H1/42 20060101
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CIPS	D06M13/372 20060101
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CIPN	D06M101/18 20060101
CIPN	D06M101/20 20060101

**RELATED-ACC-NO:** 2000-064296**ABSTRACTED-PUB-NO:** EP 516412 A2**BASIC-ABSTRACT:**

Fibres are treated to improve their lubricity and antistatic properties comprises applying a liq. lubricating finish to the surface of an extruded polyolefin-contg. fibre or filament and then mechanically processing the fibre or filament, the finish provides lubricity for the processing operations and lacks sufficient lipophilic substits. to produce significant surfactant properties. It is characterised in that the finish is selected from (1) a polyol of formula  $(R)m--C--(CH_2OH)_{4-m}$  or gp. (ii) where R is 1-4C alkyl; m is 0-3; and n is 0-4; (2) a water-soluble ester or polyester obtd. by reacting the polyol (1) with a fatty acid having up to 6C atoms in a linear or branched chain; (3) a glycol obtd. by reacting the polyol (1) with ethylene oxide.

Also claimed is the use of the finish on the fibres or filaments.

Method improves their lubricity and antistatic properties and to facilitate processing them into hydrophobic nonwoven articles. The finishes can be used as initial spin finishes or as overfinishes. The fibres can be used as hydrophobic nonwoven articles, while retaining a level of hydrophobicity that is acceptable for use as the layer of hydrophobic material in prods. used for personal hygiene that have a fluid-absorbent core and a layer of hydrophobic material that isolates fluids already absorbed in the core.

**CHOSEN-DRAWING:** Dwg. 0/0

**TITLE-TERMS:** TREAT HYDROPHOBIC POLYOLEFIN  
CONTAIN FIBRE FINISH SELECT POLY  
OL WATER SOLUBLE ESTER POLYESTER  
GLYCOL IMPROVE LUBRICATE  
ANTISTATIC PROPERTIES

**DERWENT-CLASS:** A17 A25 A87 F06

**CPI-CODES:** A04-G01E; A08-M03A; A11-C05C; A12-  
S05S; F01-H06A; F02-C01; F03-C05;

**SECONDARY-ACC-NO:**

**CPI Secondary Accession Numbers:** 1992-177743